

Abstract No. wron685

X Ray Absorption XANES and EXAFS Study of NiCo(OH)₂ Active Mass in the Nickel Positive Electrode of the Nickel Metal Hydride Cell

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Beamline: X11A

Introduction: The positive electrodes in rechargeable nickel metal hydride battery (also in NiCd cell) are a functional composite consisting of a mixture of fine nickel metal powder and cobalt-doped nickel hydroxide powders "pasted" into a porous structure of metallic nickel foam. The factors that affect the electrochemical performance of electrodes, in particular their charge acceptance and discharge capacity are determined by the microstructure of the materials used (**Ref**).

Methods and Materials: An exploratory XAS examination of two grades of fresh as well as spent rechargeable Ni hydroxide powders, and moulded electrodes was performed to compliment data being collected using XRD, XPS, FE-TEM and microcalorimetry techniques. Beam line X-11A at the NSLS was used to collect Ni and Co K-edge XANES and EXAFS spectra. The spectra were recorded using the transmission and fluorescence modes of detection.

Results: No K edge shift was observed when comparing two grades of fresh battery hydroxides which differ in their microstructure, as quantitized by the degree of disorder (DODO) in stacking of hexagonal planes revealed by either X-ray diffraction or direct lattice imaging in TEM. Although, the more disordered powder features contained broader absorption peaks. Two electrodes were analyzed after full discharge (SOC=0%). The NiK -edge position shifted to higher energy indicating that the average Ni valency increases irreversibly in spent electrodes, although the valency can be returned to low Ni (II) states in electrodes used only one cycle, or cycled until the maximum capacity was reached (100 cycles). This may be correlated with pronounced broadening of the absorption peaks and the appearance of an amorphous fraction, as being evidenced by scanning microcalorimetry.

Conclusions: . The difficulty in electrode charging (charge inefficiency) which follows may be responsible for electrode failure. Processing and interpretation of the respective chi (k) Ni K- and Co K spectra is in progress.

References: Ref. Z.S. Wronski, Int. Materials Reviews, 46 (1), 2001, p.1-52.